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Synthesis and lipase-catalyzed resolution of 5-(hydroxymethyl)-1,3-dioxolan-4-ones

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Enantiomeric excess Determination of Compounds **2a,b**, **9-12** resolved by *Candida antarctica* lipase

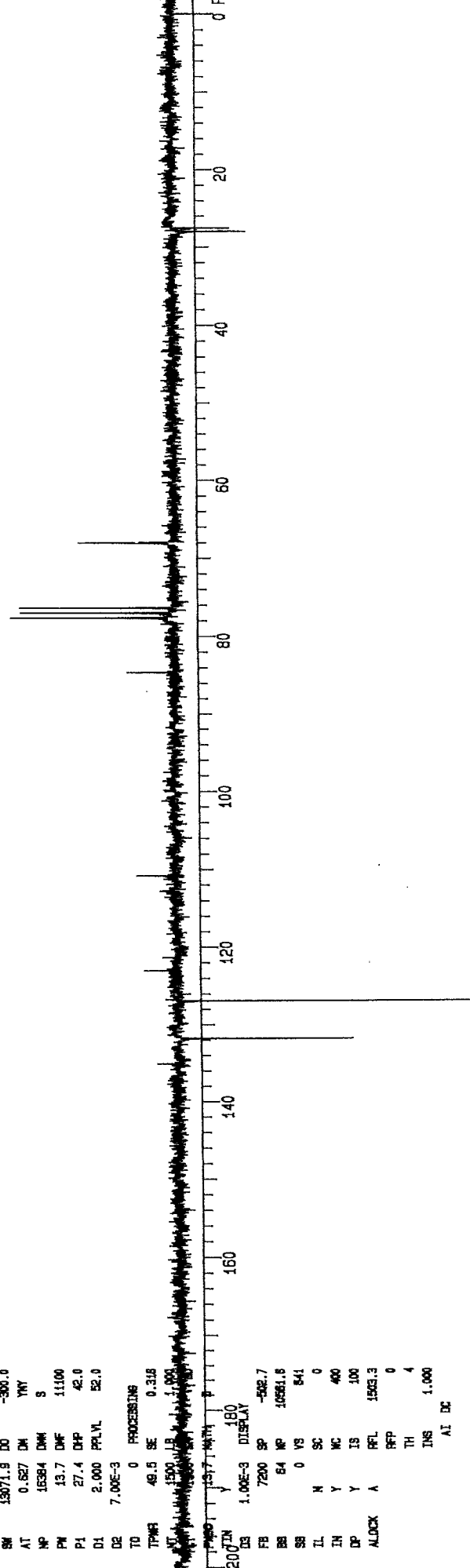
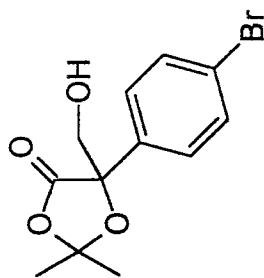
Resolved by HPLC on Daicel OJ column

Compound	Solvent	Flow rate (ml/min)	t _{R1} (min)	t _{R2} (min)
2a	hexane/ipa 8:2	1.0	7.5(major,S)	12.4(minor,R)
11a	hexane/ipa 8:2	1.0	10.6(major,R)	14.9(minor,S)
2b	hexane/ipa 8:2	0.5	13.8(major)	45.2(minor)
11b	hexane/ipa 8:2	0.5	20.7(minor)	23.6(major)
10	hexane/ipa 9:1	0.5	19.5(major)	23.3(minor)
11d	hexane/ipa 8:2	1.0	7.1(major)	16.4(minor)
12	hexane/ipa 9:1	1.0	23.5(minor,R)	37.4(major,S)

Resolved by GC on FS-LIPODEX C column

Compound	Carrier gas	Oven temp (°C)	t _{R1} (min)	t _{R2} (min)
9	He	125	164.8	172.2
11c	He	125	145.6	147.3

In general, conditions were optimized to perform the analysis of **2a,b** and the corresponding acetate **11** in a single run. Only for **10** did the components have to be separated prior to analysis by chromatography (silica, ether).

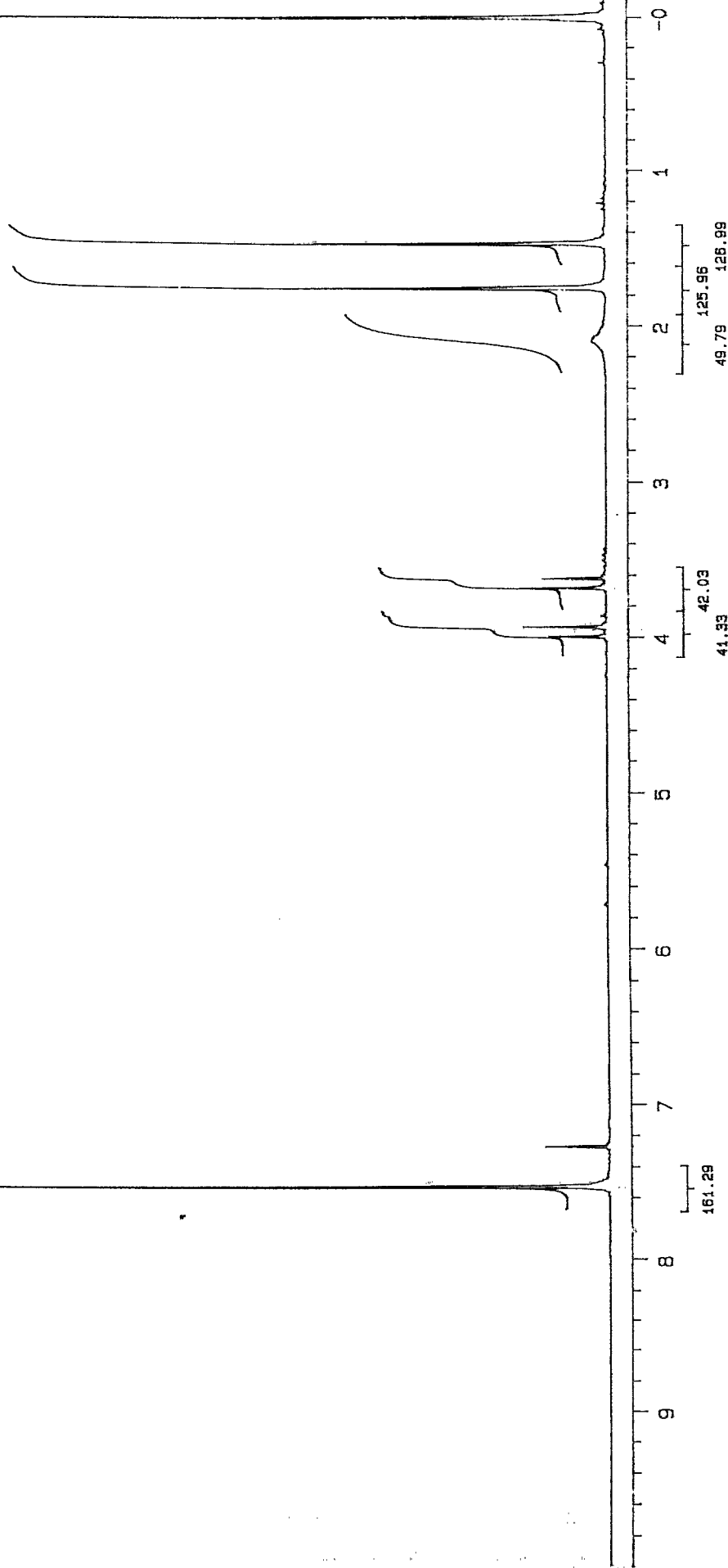
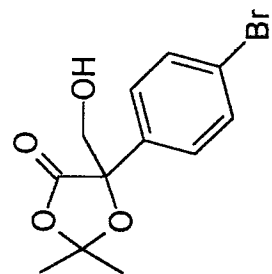


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BW	13071.9	DO	-300.0
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NP	16384	DAM	S
PW	13.7	DHF	11100
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D1	2.000	DFVL	52.0
D2	7.00E-3		
TO	0	PROCESSING	
TMR	49.5	SE	0.315
UT	1500	IS	1.000
VTN	13.7	MTH	0
FTN	13.7	MTH	0
FB	1.00E-3	DISPLAY	180
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IS	Y	IS	100
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ALOCK		RFP	0
TH		TH	4
INS		INS	1.000
AI		AI	DC

NAME: NAME
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161.29

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49.79

125.99

-0

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2

3

4

5

6

7

8

9

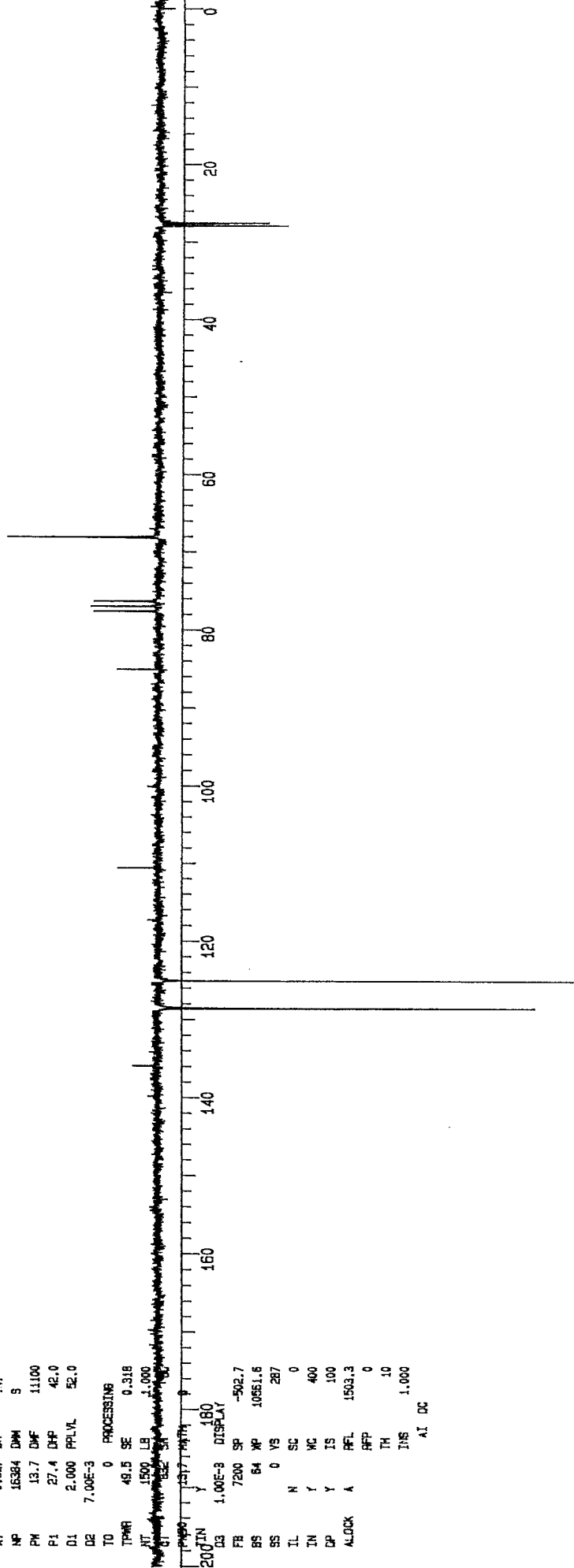
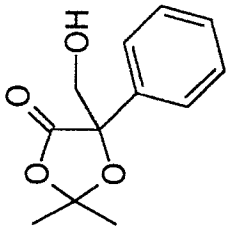
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03	3905.2	77.654	15.190
04	2973.1	77.015	16.377
05	2241.1	26.381	15.527
06	2425.4	62.132	36.102
07	1404.0	27.919	-21.079
08	1395.3	27.557	-26.995

SOLVENT C0CL3
 FILE R0802

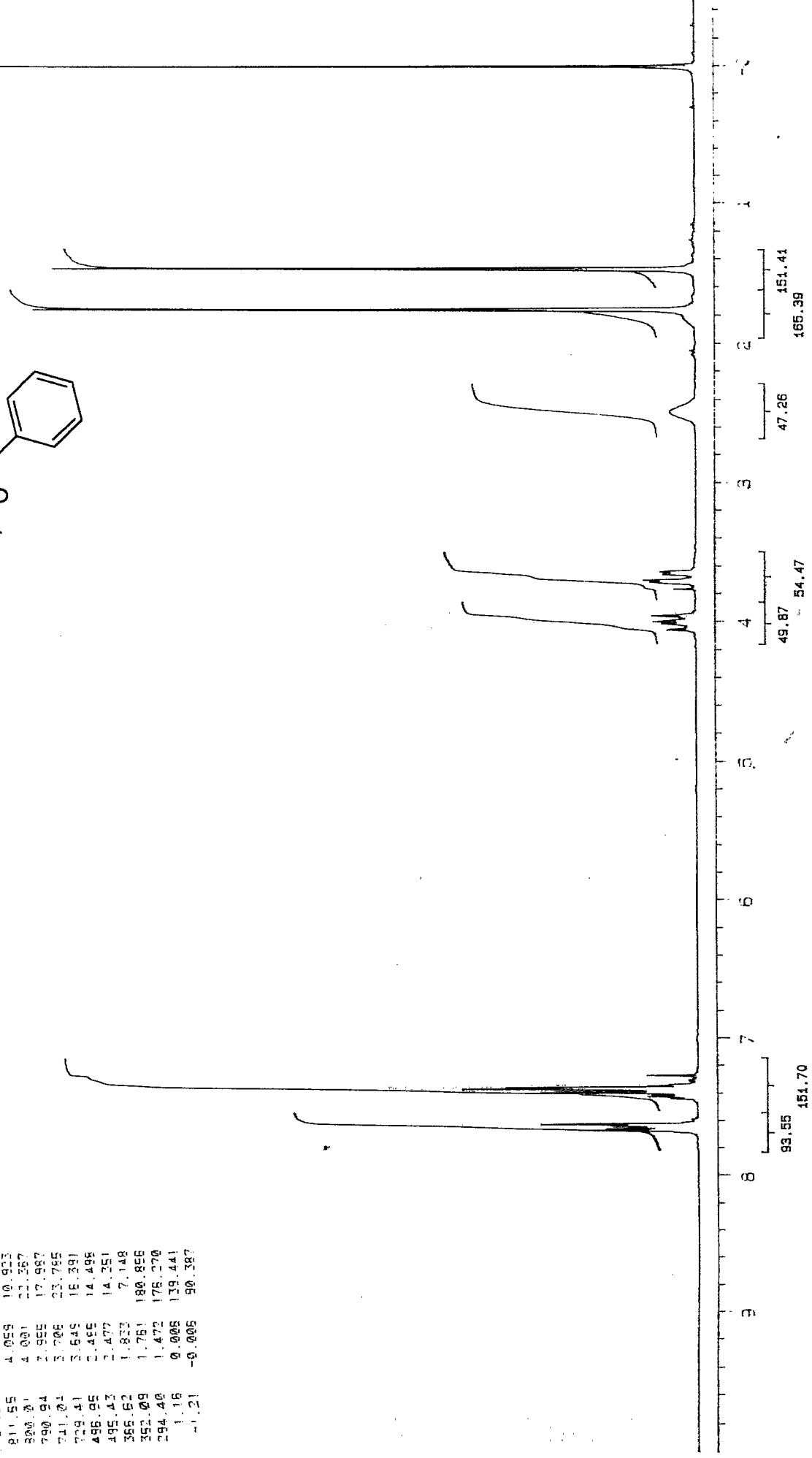
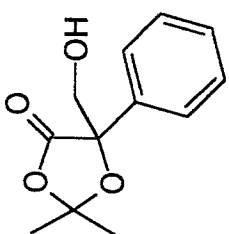
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PM	13.7 DM 1100
P1	27.4 DM 42.0
D1	2.000 PRLVL 52.0
D2	7.00E-3
TO	0 PROCESSING
TPMR	49.5 SE 0.318
AT	1500 LB 1.000
CT	1500 LB 1.000

FB	7200 SP	-502.7
BS	64 NP	10551.6
SS	0 VS	287
IL	N SC	0
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	AI DC	



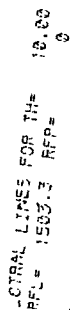
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PPM	AREA	CPM	INTENSITY
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7.639	55.572		
7.361	85.154		
7.271	8.365		
7.259	5.847		
4.059	10.923		
4.001	22.367		
3.955	17.987		
3.706	23.785		
3.649	15.391		
2.495	14.498		
2.472	14.751		
1.873	7.148		
1.761	190.856		
1.472	175.270		
0.005	139.441		
-0.005	90.387		



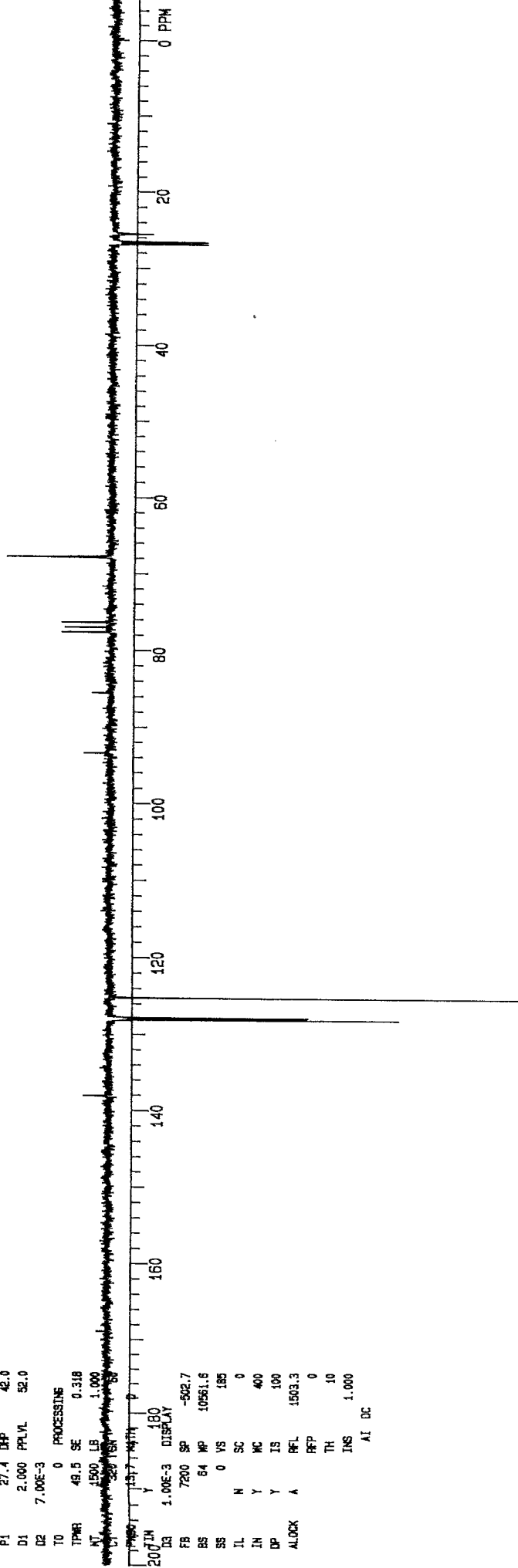
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3	732.92	3.665	9.129
4	715.50	3.573	7.325
5	710.80	3.554	10.441
6	703.77	3.519	5.338
7	699.07	3.496	5.739
8	593.10	3.366	3.116
9	586.45	2.932	8.674
10	521.23	2.306	5.453
11	398.33	1.952	4.434
12	317.14	1.566	46.304
13	312.49	1.563	105.813
14	270.56	1.353	179.688
15	265.44	1.327	18.545
16	-11.09	-0.055	10.905

ACQUISITION		DEC. \$ VT	
TH	1.000	DN	1.000
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SM	2.340	DM	NNN
MP	16384	DPP	21.0
PN	2.5	PFL	56.0
P1	0		
D1	1.000	PROCESSING	
D2	0	MATH	D
TO	0		
NT	32	DISPLAY	
CT	32	SP	-200.0
TPMR	60.0	AP	2198.7
PMR0	13.0	YS	46
PMR1	2000	SC	0
SS	0	AC	400
IL	N	IS	291
IN	Y	PFL	643.9
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		INS	1.000
		AI	DC

[illegible]

SOLVENT	water
FILE	H0FA02

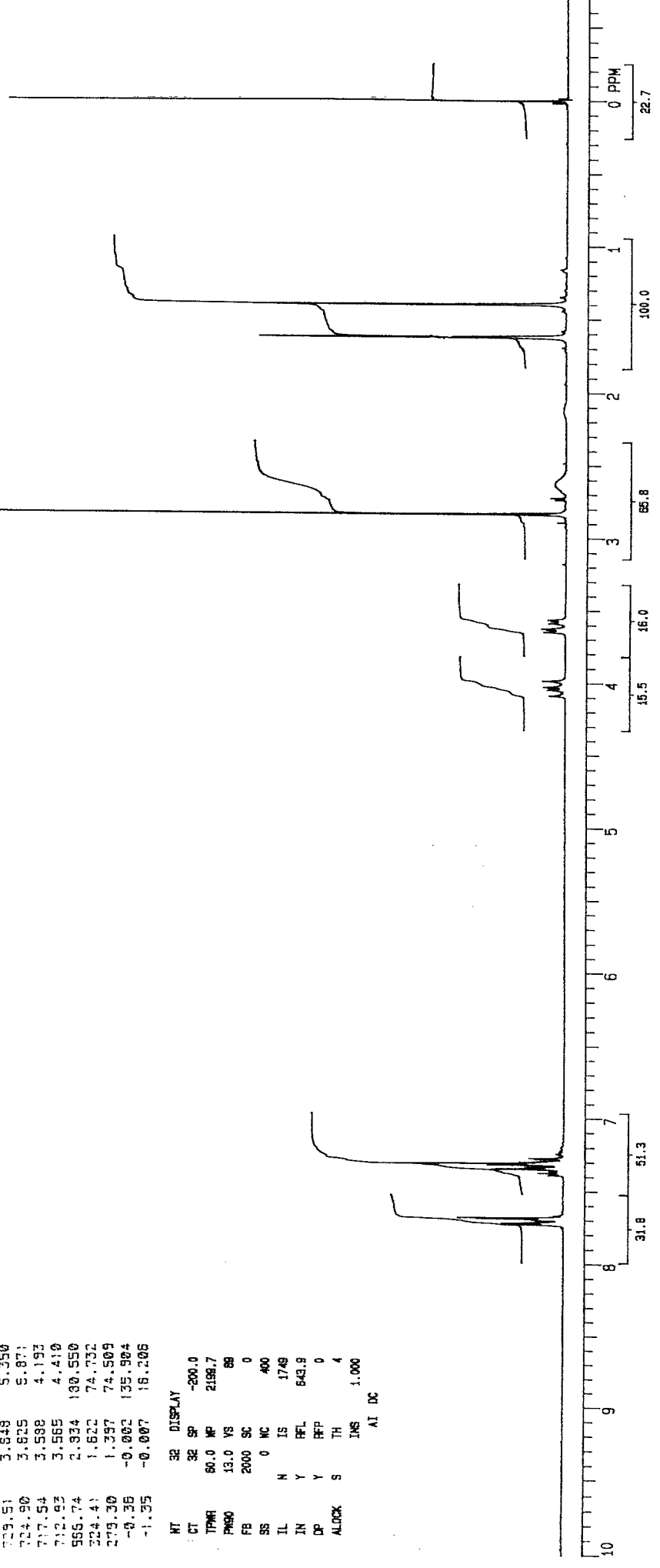
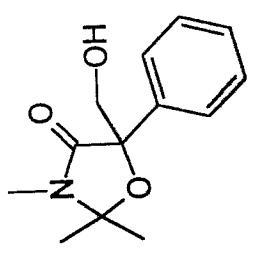
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AW	15384 DMM S			
PW	13.7 DMF	11000		
F1	27.4 DMF	42.0		
D1	2.000 PPLVL	52.0		
OZ	7.00E-3			
	TO PROCESSING			
TPMR	49.5 SE	0.318		
NF	1500 LB	1.000		
CT	320 TERN	SV		
IN	Y			
ZIN	180			
DISP	1.00E-3 DISPLAY			
FE	7200 SP	-502.7		
BES	64 MP	10951.6		
SS	0 VS	185		
IL	N SC	0		
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Y	Y TS	100		
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AI DC				



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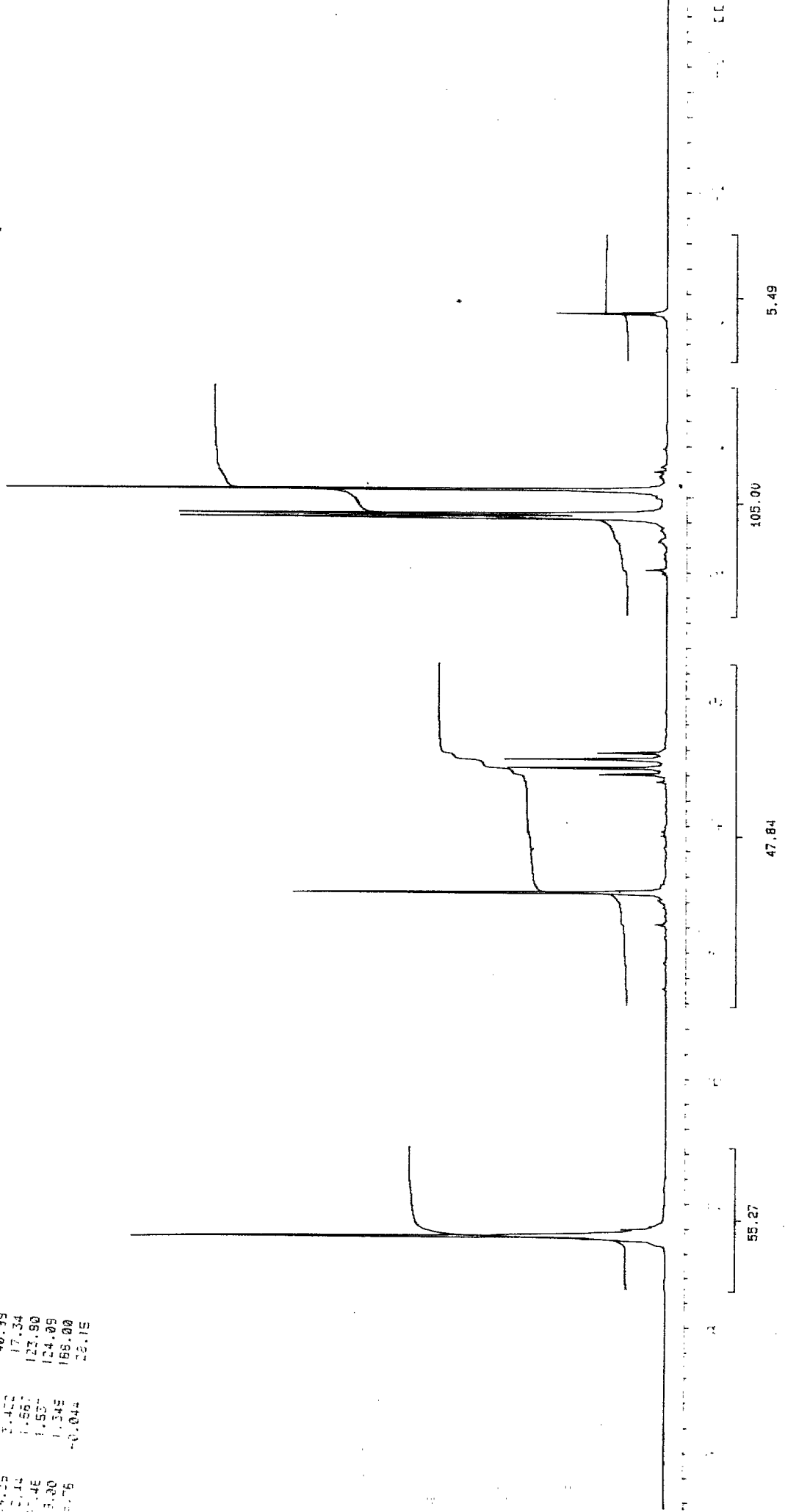
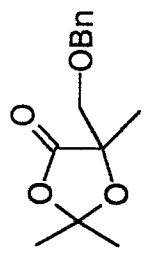
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15.231	15.231
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15.225	15.225
15.222	15.222
15.219	15.219
15.216	15.216
15.213	15.213
15.210	15.210
15.207	15.207
15.204	15.204
15.201	15.201
15.198	15.198
15.195	15.195
15.192	15.192
15.189	15.189
15.186	15.186
15.183	15.183
15.180	15.180
15.177	15.177
15.174	15.174
15.171	15.171
15.168	15.168
15.165	15.165
15.162	15.162
15.159	15.159
15.156	15.156
15.153	15.153
15.150	15.150
15.147	15.147
15.144	15.144
15.141	15.141
15.138	15.138
15.135	15.135
15.132	15.132
15.129	15.129
15.126	15.126
15.123	15.123
15.120	15.120
15.117	15.117
15.114	15.114
15.111	15.111
15.108	15.108
15.105	15.105
15.102	15.102
15.099	15.099
15.096	15.096
15.093	15.093
15.090	15.090
15.087	15.087
15.084	15.084
15.081	15.081
15.078	15.078
15.075	15.075
15.072	15.072
15.069	15.069
15.066	15.066
15.063	15.063
15.060	15.060
15.057	15.057
15.054	15.054
15.051	15.051
15.048	15.048
15.045	15.045
15.042	15.042
15.039	15.039
15.036	15.036
15.033	15.033
15.030	15.030
15.027	15.027
15.024	15.024
15.021	15.021
15.018	15.018
15.015	15.015
15.012	15.012
15.009	15.009
15.006	15.006
15.003	15.003
15.000	15.000

32 DISPLAY
 CT 32 SP -200.0
 TMR 60.0 MP 2196.7
 PMSO 13.0 VS 60
 FB 2000 SC 0
 SS 0 NC 400
 IL N IS 1749
 IN Y RFL 643.9
 DP Y RFP 0
 ALLOC S TH 4
 INS 1.000
 AT DC



Δ TOTAL LINES FOR TH = 14.35
 2105.40, REF = 1451.82

FREQ	FFA	INTENSITY
1452.66	1.129	29.76
1451.81	7.160	136.01
1406.28	4.677	94.68
1381.94	3.533	16.93
1361.23	3.542	40.21
1311.91	7.472	40.99
1294.35	3.422	17.34
1277.14	1.667	123.90
1274.46	1.537	124.09
123.80	1.345	166.00
117.76	-0.044	22.15



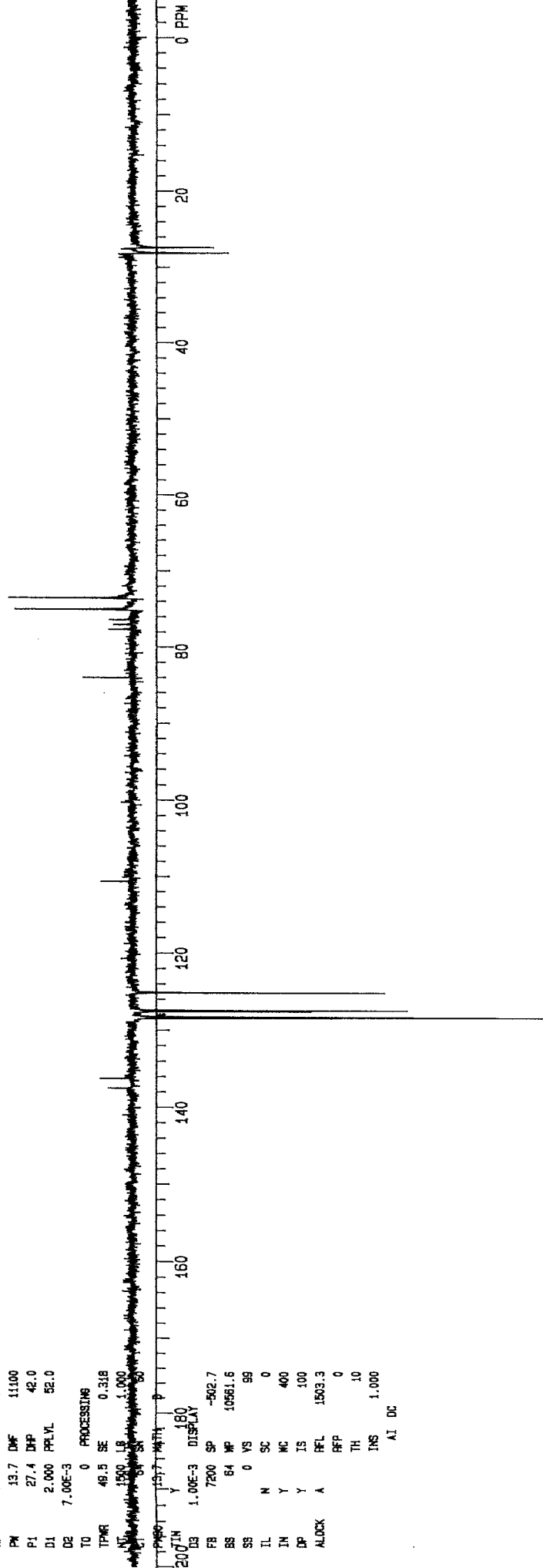
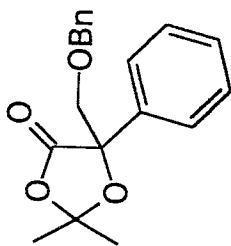
IRL LINES FOR TH= 10.00
 CF= 1500.0 PPF= 0

FREQ	PPM	INTENSITY
6455.4	123.533	-50.691
6450.0	123.458	-120.927
6455.8	123.363	-69.179
6450.0	122.888	-43.583
6450.0	122.508	-65.635
6450.0	122.202	-65.523
6450.0	121.038	12.108
6450.0	120.142	23.892
6450.0	119.242	30.242
6450.0	118.183	-23.292
6450.0	117.447	-19.534

ACQUISITION DEC. & VT

TH	13.000	DN	1.000
SM	13071.9	DO	-300.0
AT	0.827	DM	YNY
NP	16384	DM	S
PM	13.7	DM	11100
P1	27.4	DPP	42.0
D1	2.000	PPYL	52.0
D2	7.00E-3		
T0	0	PROCESSING	
TPMR	48.5	SE	0.318
UN	1500	LS	1.000
CL	1500	SS	1.000

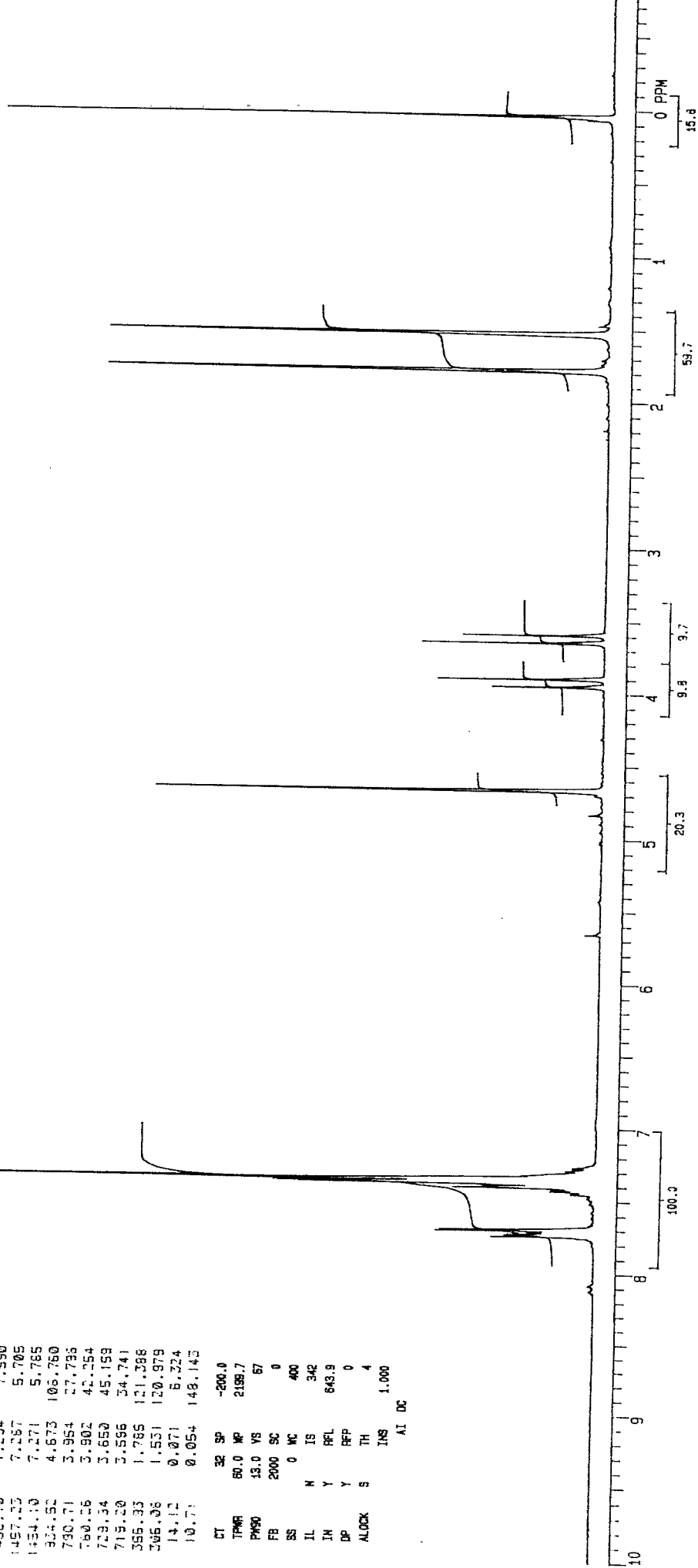
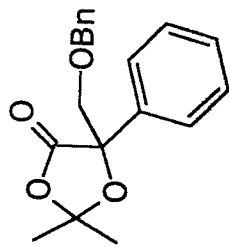
200 IN Y 180
 EQ 1.00E-3 DISPLAY
 FB 7200 SP -502.7
 BS 64 WP 10551.6
 SS 0 VS 99
 IL N SC 0
 IN Y MC 400
 DP Y IS 100
 ALOCK A RFL 1503.3
 RFP 0
 TH 10
 TMS 1.000
 AI DC



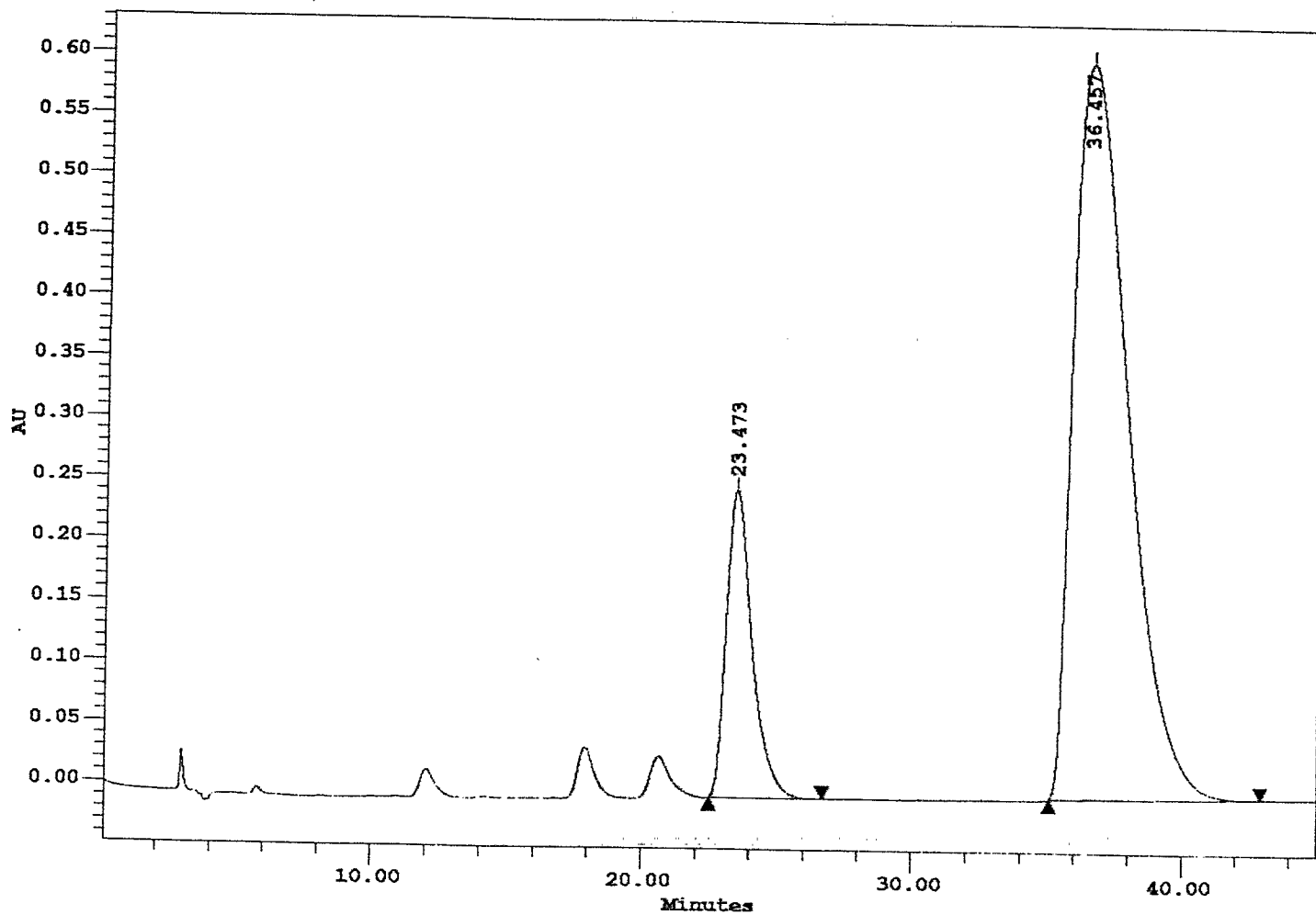
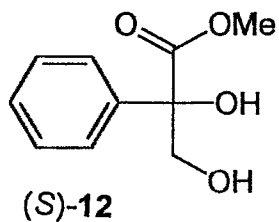
13C NMR SPECTRAL LINES FOR TH= 4.00
 REF= 543.9 APP= 0

PPM	INTENSITY
159.901	7.746
159.901	26.107
159.901	7.735
159.901	23.123
159.901	7.715
159.901	16.453
159.901	7.706
159.901	38.352
159.901	7.637
159.901	37.612
159.901	7.452
159.901	8.242
159.901	7.443
159.901	5.609
159.901	7.435
159.901	11.000
159.901	7.426
159.901	10.729
159.901	7.403
159.901	35.062
159.901	7.400
159.901	27.251
159.901	7.368
159.901	78.096
159.901	7.355
159.901	181.297
159.901	7.326
159.901	18.078
159.901	7.313
159.901	12.074
159.901	7.294
159.901	7.290
159.901	7.267
159.901	5.705
159.901	7.271
159.901	5.785
159.901	4.673
159.901	105.760
159.901	3.954
159.901	27.736
159.901	3.902
159.901	42.254
159.901	3.650
159.901	45.159
159.901	3.596
159.901	34.741
159.901	1.785
159.901	121.388
159.901	1.531
159.901	120.979
159.901	0.071
159.901	5.224
159.901	0.054
159.901	148.143

CT 32 SP -200.0
 TMR 60.0 WP 2198.7
 PMSO 13.0 VS 57
 FB 2000 SC 0
 BS 0 MC 400
 IL N IS 342
 IN Y RFL 643.9
 DP Y RFP 0
 ALOCK S TH 4
 INS 1.000
 AI DC



Structure



Peak Results

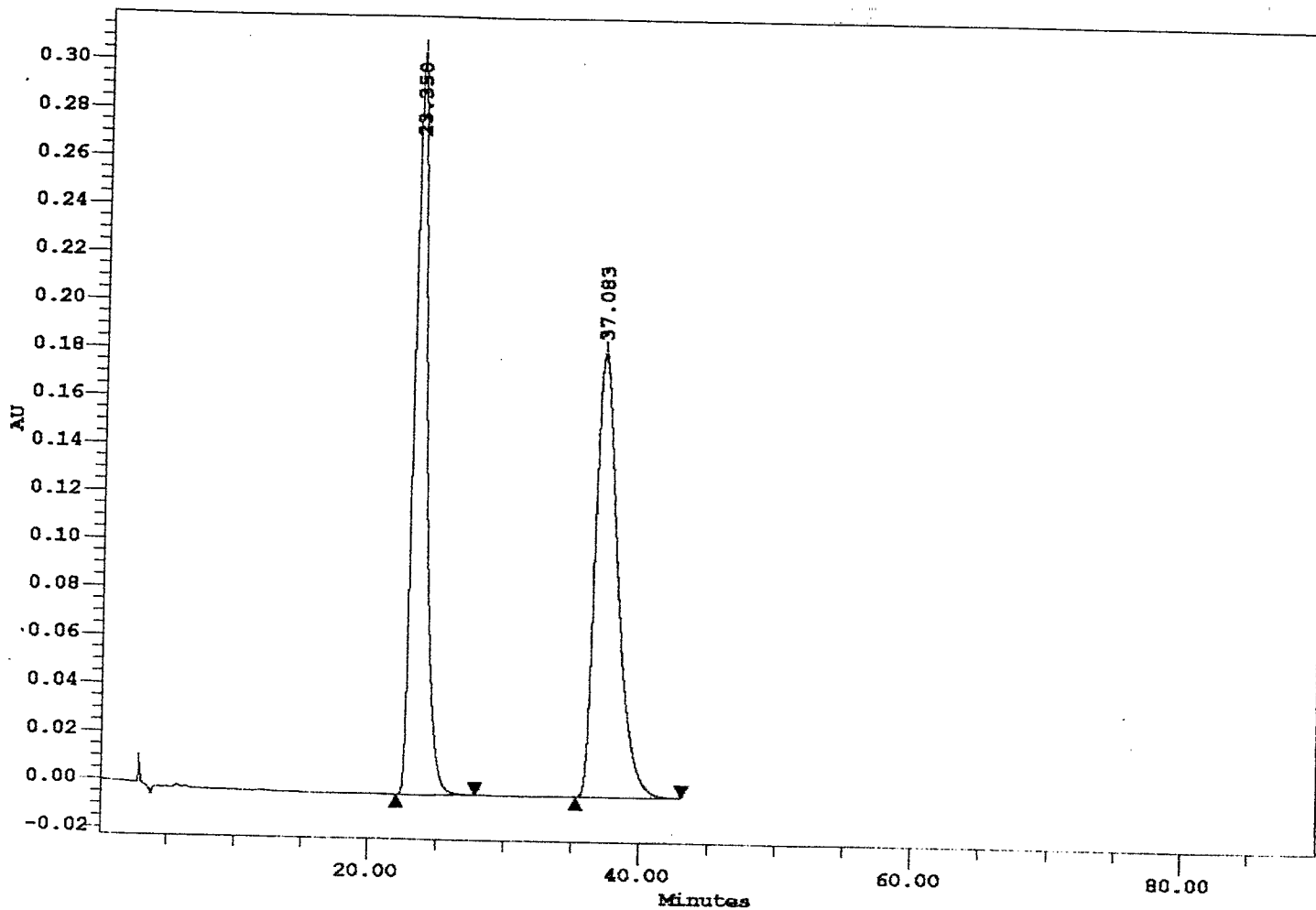
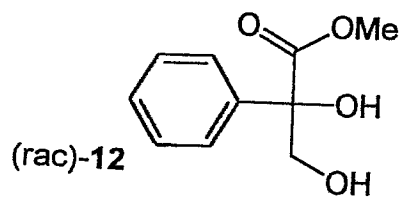
#	Name	Ret Time (min)	Area (uV*sec)	% Area	Height (uV)	Int Type
1	Peak1	23.473	16580150	16.19	252225	BM
2	Peak2	36.457	85860308	83.81	605068	BM

Column Name

Mobile Phase

Flow Rate

Structure



Peak Results

#	Name	Ret Time (min)	Area (uV*sec)	% Area	Height (uV)	Int Type
1	Peak1	23.350	21081378	49.83	308669	BM
2	Peak2	37.083	21223767	50.17	184830	BM

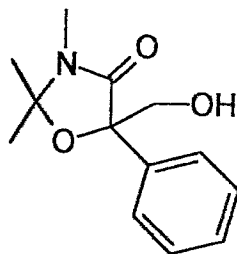
For Sample: NOH

Vial: 2 Chan: 991M

Date Processed 17/01/95 15:25

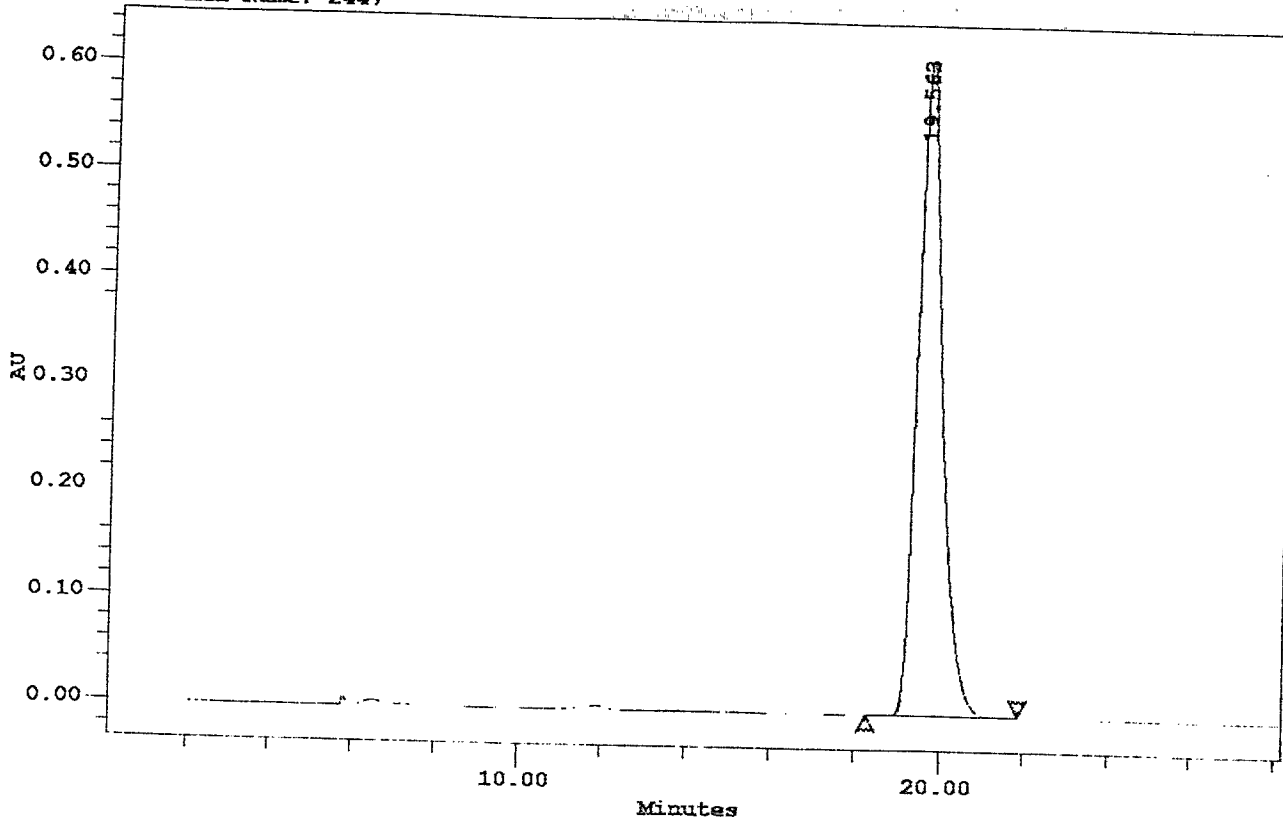
MobilePhase: 10% ipa in hex f=.5 ColName: OJ

Struc. Form.



(S)- 10 prepared by CAL resolution

Channel Name: 2447



report

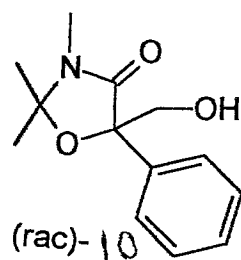
#	Name	Ret Time (min)	TR_PH	Height (uV)	Area (uV*sec)
1	Peak1	19.56	12111197	619076	25249428

For Sample: dioxolanon Vial: 4 Chan: 991M

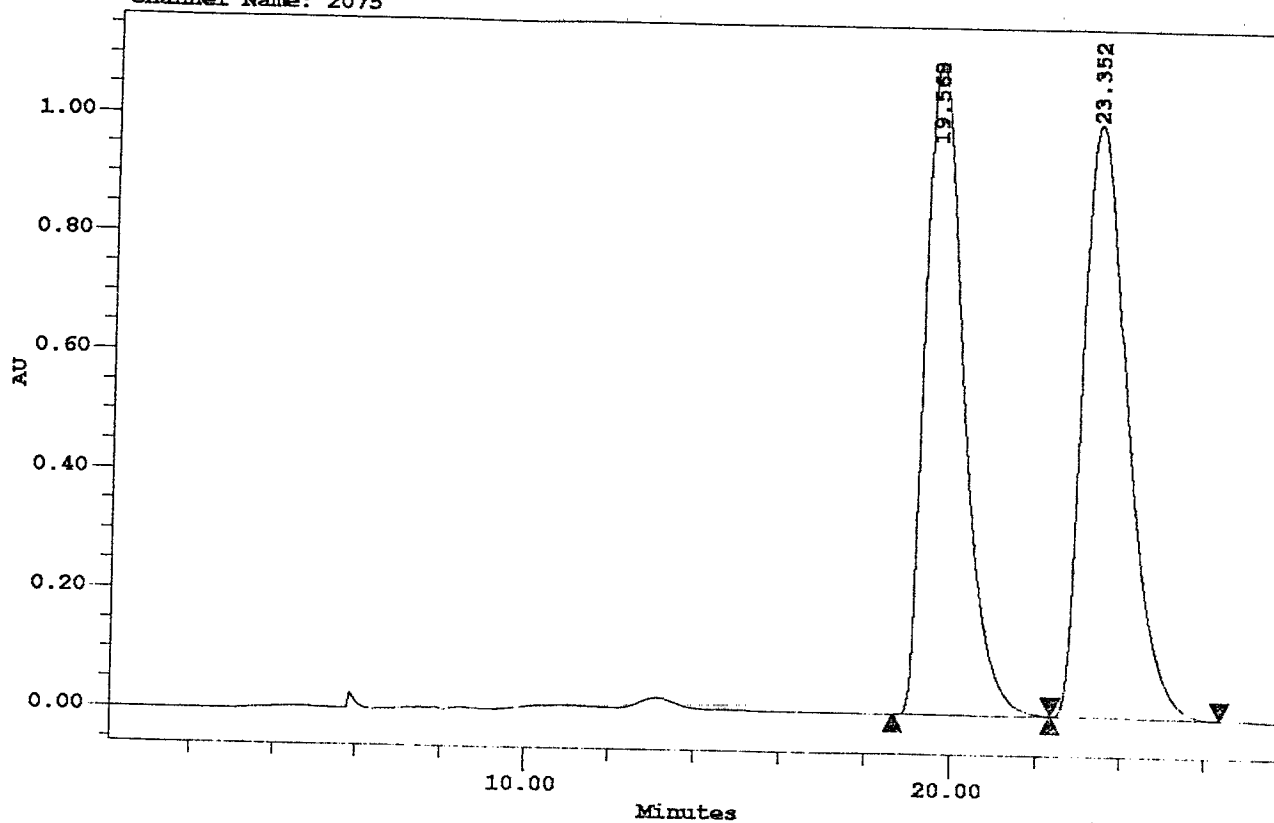
Date Processed 27/10/94 15:28

MobilePhase: 10% ipa in hex f=.5 ColName: OJ

Struc. Form.



Channel Name: 2075



report

#	Name	Reten Time (min)	TR_PH	Height (uV)	Area (uV*sec)
1	Peak1	19.57	21531031	1100300	69748908
2	Peak2	23.35	23341520	999565	73233862

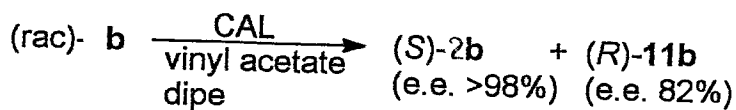
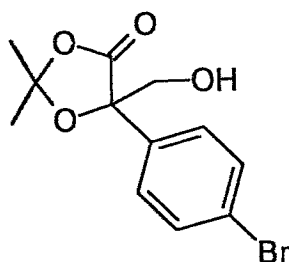
For Sample: brmethy1 5 dag Vial: 1 Chan: 991M

Date Processed 11/04/94 15:54

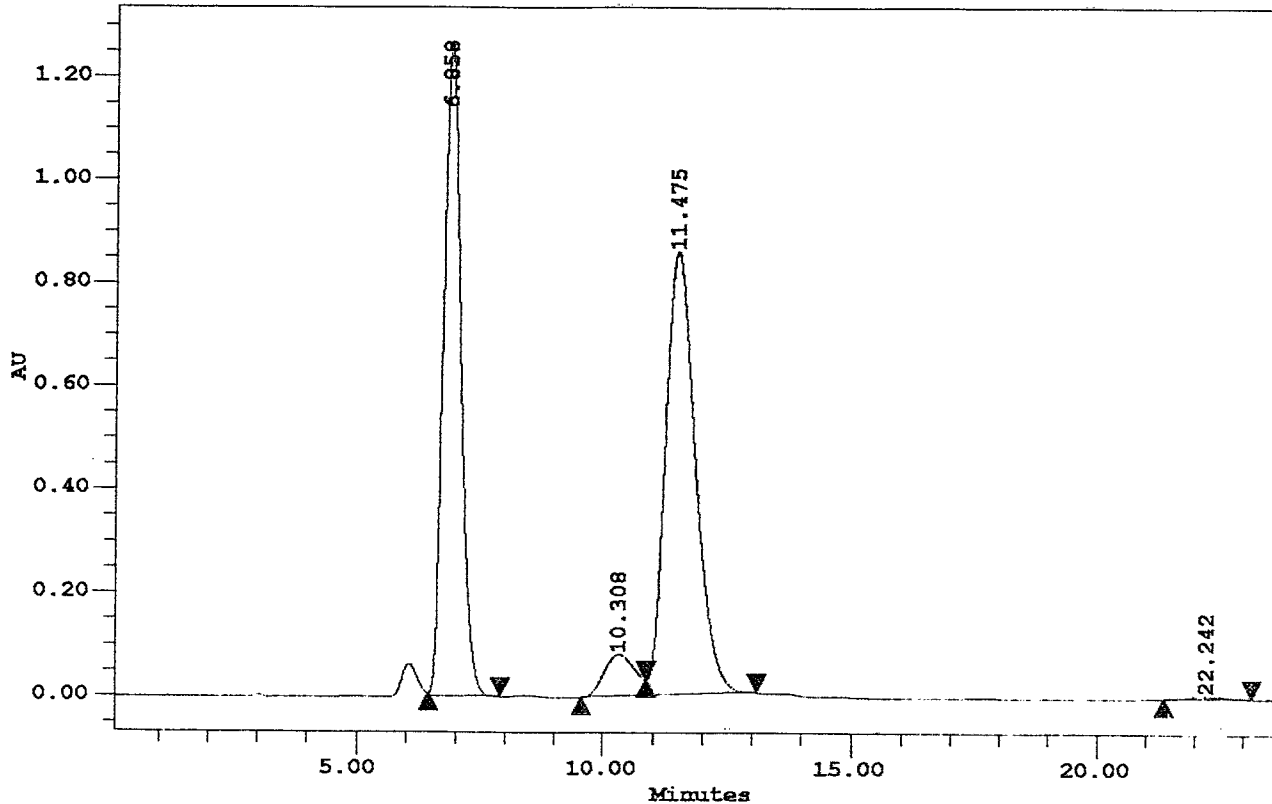
MobilePhase: 20% ipa in hexane

ColName: oj

Struc. Form.



Channel Name: Wvln Ch1



report

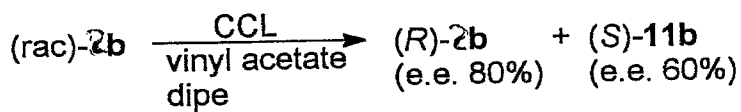
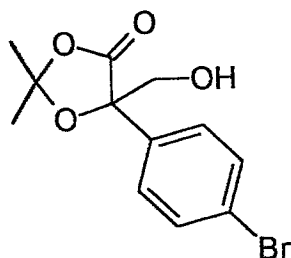
#	Name	Reten Time (min)	TR_PH	Height (uV)	Area (uV*sec)
1		6.86	0	1270234	30846958
2		10.31	0	81251	3520144
3		11.48	0	857834	35910466
4		22.24	0	6336	348488

For Sample: p-brCH₂OH CC Vial: 1 Chan: 991M

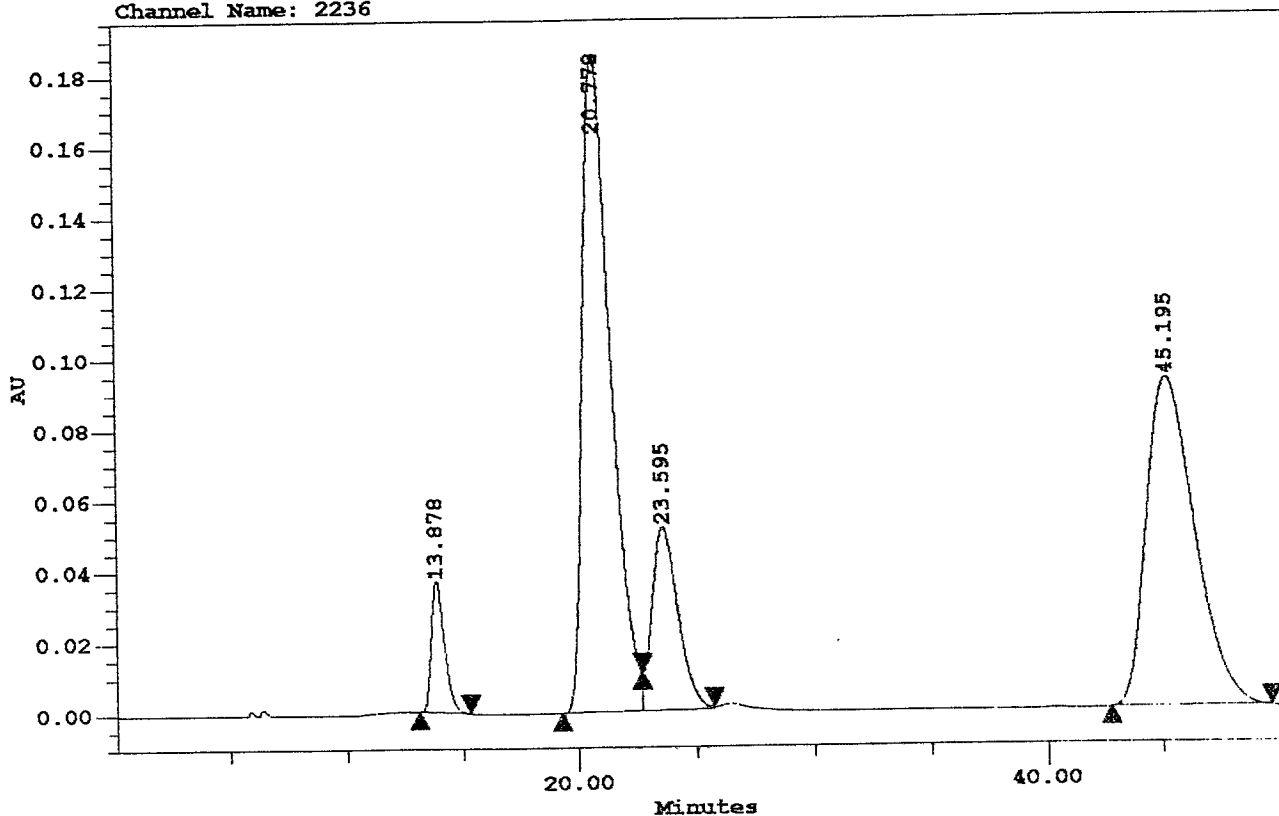
Date Processed 07/04/94 13:12

MobilePhase: 20% ipa in hexane ColName: OJ

Struc. Form.



Channel Name: 2236



report

#	Name	Rete Time (min)	TR_PH	Height (uV)	Area (uV*sec)
1	Peak1	13.88	512647	36939	1459470
2	Peak2	20.78	3861037	185820	15491997
3	Peak3	23.59	1221203	51757	3934900
4	Peak4	45.19	4182736	92549	13360509

